Historic, archived document

Do not assume content reflects current scientific knowledge, policies, or practices.

SHOUT
SHARE
IT

UNITED STATES DEPARTMENT OF AGRICULTURE

BULLETIN No. 309

Contribution from the Bureau of Plant Industry
WM. A. TAYLOR, Chief

Washington, D. C. PROFESSIONAL PAPER November 4, 1915

ZACATON AS A PAPER-MAKING MATERIAL.

By CHARLES J. BRAND, *in Charge*, and JASON L. MERRILL, *Assistant Chemist, Paper-Plant Investigations*.[1]

CONTENTS.

	Page.		Page.
Introduction	1	Chemical investigation of the grass and pulp.	15
Botanical history and systematic position of zacaton	3	Cellulose from zacaton	18
Distribution of zacaton	6	Semicommercial tests of the pulp	20
Laboratory tests of pulp production	10	Physical tests of zacaton papers	25
Micromeasurements of fibers and other cells.	14	Conclusion	27

INTRODUCTION.

There appears to be a constant and increasing interest in the discovery of plant materials which may be substituted for wood and rags in the making of paper stock of various kinds. The uses to which paper may be put are multiplying rapidly, the consumption for present purposes is increasing greatly, and there is a constant depletion of existing supplies. Many materials from both wild and cultivated plants are at present going to waste, so that a natural desire to save them adds to the general interest in the subject. This interest is world wide and practically spontaneous. In southern China bamboos and rice straw are under experiment; in Manchuria the stalks of the grain sorghums; in Mexico wood waste and various trees not now used for other purposes; and in Egypt the plant formation known as Nile suud, which constitutes the dense jungle growth of the upper White Nile and contains a large proportion of papyrus plants. In the Philippines attention is being given to bamboos and various other grasses and also to the fibrous by-products of the Manila-hemp indus-

[1] The Paper-Plant Investigations of the Bureau of Plant Industry are conducted under the direction of Charles J. Brand, Chief of the Office of Markets and Rural Organization.

NOTE.—This bulletin should be useful to all persons who are interested in the economic phases of paper making, especially to print and book paper manufacturers. It has a botanic and chemical interest as well.

6826°—Bull. 309—15——1

try. In this country scarcely a month passes during which some new wild plant or crop waste is not proposed as a certain and permanent relief to the paper manufacturer from the stress resulting from the rising cost of raw materials.

The past 10 years have witnessed an enormous growth in the pulp and paper industry and a keener realization of the fact that the present wood supply of the United States can not indefinitely withstand the demands placed upon it. About 80 per cent of the paper stock used in this country is derived from wood. In 1900 about 2,000,000 cords of wood were used for pulp manufacture, and the present use is approximately 4,500,000 cords a year. Pulp-wood imports in this country increased from 650,000 cords at $4.20 per cord in 1907 to 1,036,000 cords at $6.60 in 1913. In 1903, 131,000 tons of wood pulp were imported, as against 563,000 tons in 1913.

In a report of the United States Forest Service in 1914 the annual growth of wood in the United States is placed at 12 cubic feet per acre per year, while there are being removed 36 cubic feet per acre per year; in other words, as a nation, wood is being used three times as fast as it grows. Without doubt imported wood will play an important rôle in the paper industry of this country for many years to come.

New woods are in common use to-day which would not have been considered a few years ago, and reforesting is being given very serious attention, all of which goes to show a desire on the part of the pulp manufacturer to husband his present source of supply or to secure new sources.

Since the demand for paper stock is gaining so rapidly upon the supply it is very clear that the price of raw material will continue to increase and in so doing will bring other raw materials into competition. It is for this reason that investigations of the adaptability of fibrous plants and crop wastes should be carried on with some of the more promising materials.

The Office of Paper-Plant Investigations of the Bureau of Plant Industry has numerous materials under examination and proposes from time to time, as the data obtained may warrant, to publish the information which has been secured. The publication of these data will not mean that the work with the material has been completed or that the conclusions reached are final. There is always a possibility that further information and the devising of new and better methods may result in taking a raw material from the class of unpromising materials and placing it in the class of promising materials.

The work with zacaton (*Epicampes macroura* Benth.) has progressed to a point where at least a preliminary publication of results is desirable.

BOTANICAL HISTORY AND SYSTEMATIC POSITION OF ZACATON.

The genus Epicampes was established by Dr. J. S. Presl, of the University of Prague, in his treatment of the Gramineæ in 1830.[1] The type species of the genus is *Epicampes strictus*, which Presl figures on plate 39 of his work.

The following characterization of the genus is taken from Scribner:[2]

Epicampes Presl, Rel. Haenk. 1:235, t. 39. 1830. Spikelets small, 1-flowered. Empty glumes 2, membranaceous, slightly unequal, convex on the back, carinate, often finely 3-nerved; flowering glumes 3-nerved, obtuse or emarginate, a little shorter or about the length of the empty glumes, and tipped with a slender, usually rather short awn, which is rarely wanting. Stamens 3. Styles distinct, short; stigmas plumose. Grain included within the glumes, free. Tall, perennial grasses with usually very long, spikelike, many-flowered panicles.

The genus belongs to the tribe Agrostideæ of Engler and Prantl,[3] to which the true esparto, *Stipa tenacissima* L., also belongs. This grass is extensively used for paper making in the Old World, the raw material coming chiefly from Spain, Algeria, and Tripoli. The species *Epicampes macroura* [4] has received several common names, most of which refer to the utilization of its roots in the manufacture of brushes. Broom-root grass, wire-grass, and rice-root grass are the common English names. Rice, in this case, has no relation to the well-known rice grain of commerce, but the name arises from the

---

[1] Presl, K. B. Reliquiae Haenkeanae . . . v. 1, p. 235, pl. 39. Pragae, 1830.

[2] Scribner, F. L. American grasses—III. U. S. Dept. Agr., Div. Agros. Bul. 20, p. 75, 1900.

[3] Haeckel, Ernst. Gramineæ (echte Gräser). *In* Engler, Adolf, and Prantl, K. A. E. Die Natürlichen Pflanzenfamilien . . . T. 2, Abt. 2, p. 45, 50. Leipzig, 1887.

[4] This grass was first brought to the senior writer's attention in December, 1909, by Mr. L. H. Dewey, Botanist in Charge of Fiber Investigations, who transmitted a bundle of the grass tops for possible test. The sample weighed between 2 and 3 pounds and had been sent to Mr. Dewey from Mexico by the Ox Fiber Brush Co., of Frederick, Md. Subsequently, Mr. O. F. Cook, Bionomist in Charge of Crop Acclimatization and Adaptation Investigations, directed the writer's attention to certain notes of his on Epicampes previously published. (Cook, O. F. Vegetation affected by agriculture in Central America. U. S. Dept. Agr., Bur. Plant Indus. Bul. 145, p. 19–20, 1909.) These notes are of sufficient interest, showing the size, resistance, and aggressiveness of the grass, to warrant quoting them in this connection. Discussing the distribution of pines and oaks as determined by the clearing of land, Mr. Cook says:

"Ability to resist fire is the characteristic that enables the pines to establish themselves in open grass lands. Young pines with the growing bud surrounded by many green needles can survive fires that kill seedlings of other plants. As the trees grow larger they are protected by a thickened bark which is a very poor conductor of heat and not readily combustible. Nevertheless, the survival of the pines depends on the chance of frequent fires which prevent the accumulation of grass in large quantities. With grass enough to burn, even large pines may be killed by fire and the pine forest driven back from areas it has already occupied. In this way a species of wire-grass (Epicampes) is destroying forests of alders and pines on the upper slopes of the Vulcan de Agua in Guatemala. Before the access of fires this grass appears to have been confined to the crater and to the very dry upper slopes, where the pine trees are small and scattering. Now that the belts of humid forests lower down have been broken by clearings the grass has the assistance of fire and is destroying the trees with increasing rapidity.

"There are no springs or streams on the upper slopes of the volcano, so that the grass is not pastured. Its long wiry stems and leaves accumulate until there are quantities of fuel sufficient to kill large trees and to drive back the forest for long distances at each conflagration. The lower the grass comes the more luxuriant its growth and the more destructive the next fire. This will continue as long as the grass is ungrazed or care is not taken to burn the grass every year in order to prevent the accumulation of dangerous quantities of fuel.

"The roots of this grass are well protected from the fire by masses of the closely packed stems. These tufts remain wet while everything else is thoroughly dried. Except in rainy weather, no water can be obtained from the extremely coarse and loose volcanic ashes and rocks of which the upper parts of the mountain are composed."

fact that the Mexican name for the roots is "Raiz de zacaton"; that is, roots of grass, in literal translation. Zacaton [1] is the name most commonly applied to the species in Mexico. The French name for the root-brush material is "chiendent," while "Mexican whisk" is still another name applied to it.

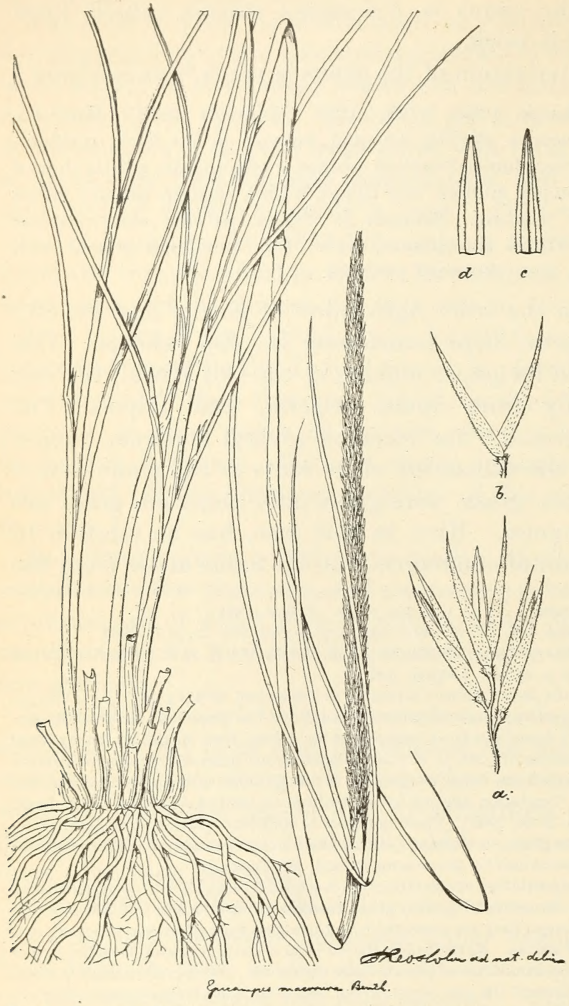

FIG. 1.—Zacaton (*Epicampes macroura*), whole plant, one-sixth natural size. *a*, Three spikelets, 4 times natural size; *b*, empty glumes, 4 times natural size; *c*, flowering glume, dorsal view, 4 times natural size; *d*, palet, dorsal view, 4 times natural size.

The first known collection of *Epicampes macroura* was made by Humboldt and Bonpland in the mountains of Toluca, in the State of Mexico, at an altitude of 10,500 feet, sometime prior to 1815. In the working up of this collection the specimens were assigned to the genus Crypsis Ait. under the specific names *macroura*, *phleoides*, and *stricta*. About 1829, when Kunth published that portion of his "Révision des Graminées" [2] containing the Agrostideæ, he evidently had changed his mind as to the assignment of these specimens to Crypsis and reassigned them to Linnæus's genus Cinna. Many years later, about 1886, Eugène Fournier,[3] in working up the collections of Mexican plants deposited in the herbarium of the Museum of Paris, established a new genus,

[1] Zacaton as a common name is applied also to *Muhlenbergia distichophylla* (Presl) Kunth and *Sporobolus wrightii* Munro.

[2] Kunth, K. S. Révision des Graminées. 3 v. Paris, 1829.

[3] Fournier, Eugène. Mexicanas Plantas . . . pars. 2, p. 90. Parisiis, 1886.

The legend of figure 2 should read:

Fig. 2. Cross section of a leaf blade, x 240, showing hypodermal stereome, the large water-storage tissue, the palisade tissue, and the mestome surrounded by a parenchyma sheath and a thick-walled mestome sheath.

The legend of figure 6, page 8, should read:

Fig. 6. Cross section of part of a culm from the epidermis to the central cavity, x 320, showing hypodermal stereome, two mestome strands, and colorless parenchyma between them.

which he called Crypsinna, based in part on the Crypsis of Humboldt, Bonpland, and Kunth,[1] and in part on the Cinna of Kunth. As established by Fournier, Crypsinna included the species *stricta*, *macroura*, and *setifolia*. In November, 1881, Bentham,[2] having seen a proof or a copy of Fournier's work before its publication, disagreed with him and assigned the species *macroura* and *stricta* both to *macroura* in the genus Epicampes Presl.

The following characterization of *Epicampes macrôura* has been translated from the original Latin description by Humboldt, Bonpland, and Kunth: [1]

Crypsis macroura. Culm erect, simple, glabrous; leaves and sheaths scabrous; panicle spikelike, very long, cylindrical, erect; glumes equal, [floret] nearly as long as the glumes. Found on the sunny side of a mountain in the State of Mexico near the mountain of Toluca, at an altitude of 11,000 feet. Flowers in September.

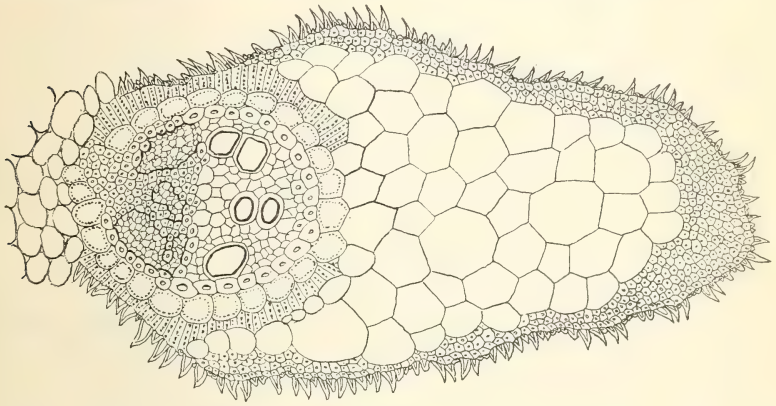

FIG. 2.—Cross section of part of a culm from the epidermis to the central cavity, × 320, showing hypodermal stereome, two mestome strands, and colorless parenchyma between them.

Culms erect, simple, 3 or 4 feet tall, glabrous, pubescent below the glabrous nodes. Leaves narrowly linear, convolute (?), striate, slightly scabrous. Sheaths rather lax, striate, glabrous, longer than the internodes. Ligule very long, less than 1 inch, bifid (2-cleft), glabrous, the lobes acuminate. Panicle spikelike, dense, cylindrical, strictly erect, 1 foot long. Spikelets pediceled, the pedicels scabrous. Glumes linear, acuminate, carinate, subequal, nearly glabrous, ciliate-hispid on the back, green. Floret slightly shorter than the glumes, lanceolate, acute, the lemma and palea concave, equal, slightly scabrous, green; the lemma 3-nerved, the palea 2-nerved, narrower.

The general appearance of a few culms taken from the tufts in which this grass usually grows is shown in figure 1, which was drawn from a herbarium specimen. Figures 2 to 6, inclusive, show the microscopical structure of the zacaton plant.[3] The appearance of an average and two small-sized tufts is shown in figure 7 These

[1] Humboldt, Alexander, Bonpland, A. J. A., and Kunth, K. S. Nova Genera et Species Plantarum . . t. 1, p. 140–141. Lutetiæ Parisiorum, 1815.

[2] Bentham, George. Notes on Gramineæ. *In* Jour. Linn. Soc. [London], Bot., v. 19, p. 87–88. 1881.

[3] The drawings for figures 1, 2, 3, 4, 5, 6, and 11 were made by Dr. Theodor Holm.

tufts furnished the raw material for the first laboratory experiments with zacaton.

DISTRIBUTION OF ZACATON.[1]

The genus Epicampes is exclusively American. About 16 species have been described, some of which, in the opinion of expert agrostologists, would not retain specific rank under critical study. The ranges of the various species extend from California and Texas southward to the Argentinian Andes Mexico is richest in number of species, and there also the root-harvesting industry has reached its highest development. *E. macroura* has been reported from many widely separated localities from Texas to Central America. The collection in the United States National Herbarium embraces specimens from the following localities in Mexico: Canyon de San Diego, State of Chihuahua; San Luis Potosi, State of San Luis Potosi;

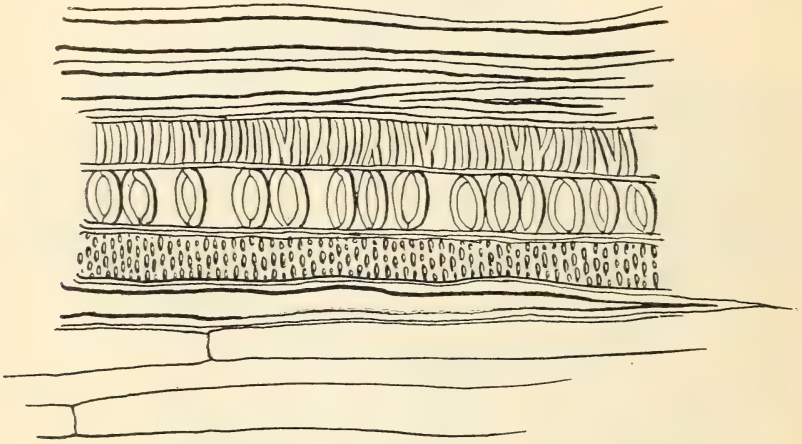

FIG. 3.—Longitudinal section of culm, × 480, showing spiral and porous vessels, stereome, and thin-walled parenchyma.

Sayula, State of Jalisco; Morelia, State of Michoacan; Nevada de Toluca, Ixtaccihuatl, Popocatepetl, Salazar, Cima, Federal District of Mexico; Eslava, State of Mexico; Mount Orizaba, San Marcos, San Andres, and San Miguel, State of Puebla.

Zacaton grows most profusely in the mountain regions east and west of the City of Mexico. It is especially luxuriant in the districts around Sayula and Toluca, in the States of Jalisco and Mexico, respectively (it will be remembered that the original collection of Humboldt and Bonpland was made on the mountain of Toluca), while the finest quality of roots is now said to be harvested around Uruapan, in the State of Michoacan. The grass is generally considered a pest, but a few attempts to subject it to crude methods of cultivation are reported to have given good results. It is perennial,

[1] Many of the data in this and the following paragraphs regarding distribution, climate, and the harvesting of the roots have been secured from Mr. A. McEwen, Frederick, Md.

and after the rainy season sends up new shoots profusely. These are relished by cattle while the tops are immature. Soon, however, the tops become so tough that stock refuse to eat them. The growth is almost entirely a wild one from self-sown seed. The mature panicles are not unlike those of timothy. Unless checked by fire, cultivation, or the harvesting of the roots, rice-root grass soon covers a field solidly. It is not uncommon to find areas many square miles in extent covered densely with this wild grass. One of the fields harvested by Mr. McEwen was 3 miles wide and 7 miles long, covered almost entirely by a relatively pure stand of *Epicampes macroura*.

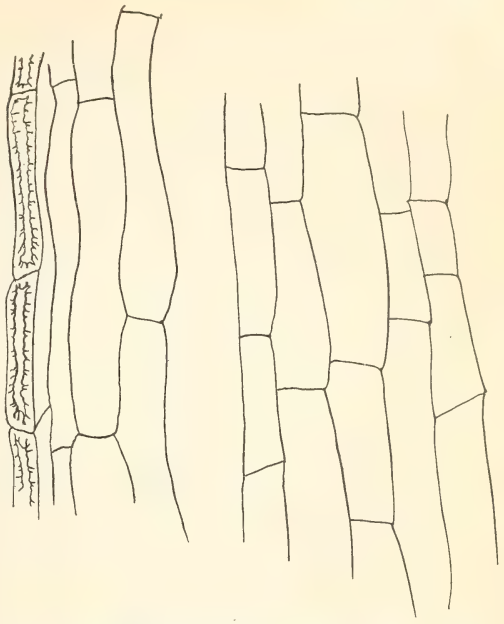

FIG. 4.—Longitudinal section of a culm, × 343, showing sclereids and pith.

The information that has been secured indicates the possibility of growing this grass successfully in some localities in the Southwest, especially for paper-producing purposes. Three allied species grow scatteringly from Texas to California.

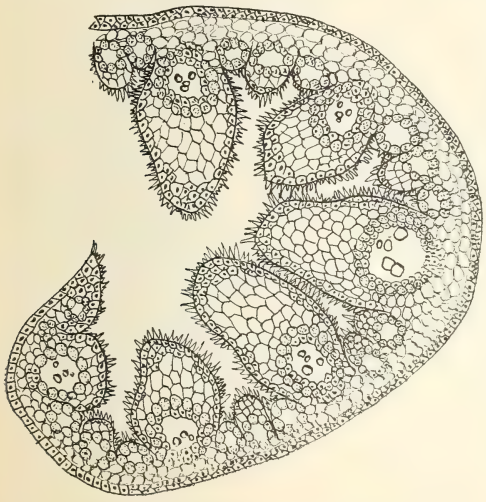

FIG. 5.—Cross section of a leaf blade, × 80.

Regarding the climate that prevails in the sections where zacaton-root harvesting is extensively carried on, Mr. McEwen states:

> We have no means of determining the rainfall, but there is a considerable quantity of rain, and the morning dew is almost as heavy as the average small shower in the States. In Sayula it rains about three months of the year, the rest of the year being dry, and one of the most beautiful climates that you can possibly imagine. The rains, when they come, are very heavy and in the middle of the day the thermometer registers about 80° F., but it is not uncommon for it to drop to 50° at night; this is a good average through the

summer, for my brother says that all through the present summer it has been cold enough for an overcoat at night in the town of Sayula, where he resides. During January, he says that it reaches to near zero and at 10 o'clock the next day it is up to 70° again.

Mr. O. F. Cook corroborates Mr. McEwen's observations regarding the relative coolness of the climate in which this grass thrives. In Guatemala, where the former has noted the plant especially, he found the same conditions.

Figure 8 shows a comparatively sparse stand of zacaton on the Vulcan de Agua, near Antigua, Guatemala, one of the early localities from which the plant was collected.

Figure 9, from a photograph taken in Guatemala, shows the grass promptly claiming the neglected portion of a formerly cultivated field on a terraced hillside along the road between Totonicapam and Quezaltenango, Guatemala, showing also in the middle distance, on the mountain slope below the pines, a characteristic wire-grass formation contending with the pines for supremacy. Both of these figures are from negatives made under the direction of Mr. Cook by Mr. C. B. Doyle, of the Bureau of Plant Industry.

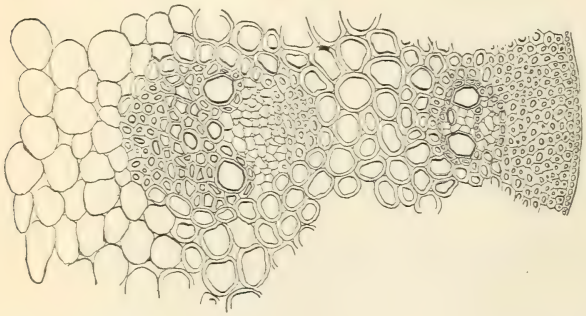

FIG. 6.—Cross section of a leaf blade, × 240, showing hypodermal stereome, the large water-storage tissue, the palisade tissue, and the mestome surrounded by a parenchyma sheath and a thick-walled mestome sheath.

The grass is said to flower from August to October, depending upon altitude and other conditions, and usually attains a height of 5 to 7 feet. The usable portions of the roots vary in length from 2 to 30 inches. The diameter of the roots range from one sixty-fourth to three thirty-seconds of an inch. They are gathered at all seasons of the year, peons digging them up with an implement resembling a hoe in shape. After washing, cleaning, and drying, the roots are cut from the grass, graded, and separated according to quality, length, and color, and finally baled ready for shipment. Vera Cruz and Tampico are the chief exporting ports, while France, Germany, and the United States are the chief users of the brushes into which the roots are manufactured. Roots of a pale yellow, a decidedly characteristic color, are preferred by the trade. It is estimated that an acre of grass yields a ton of marketable roots and at least 3 tons of tops. At present the tops are not used in any way. It seems likely that root operators might find it worth while to attempt the utilization

of the grass for pulp manufacture in sections where there are large acreages of luxuriant growth and where the cost of collecting the raw material in commercial quantities is not prohibitive.

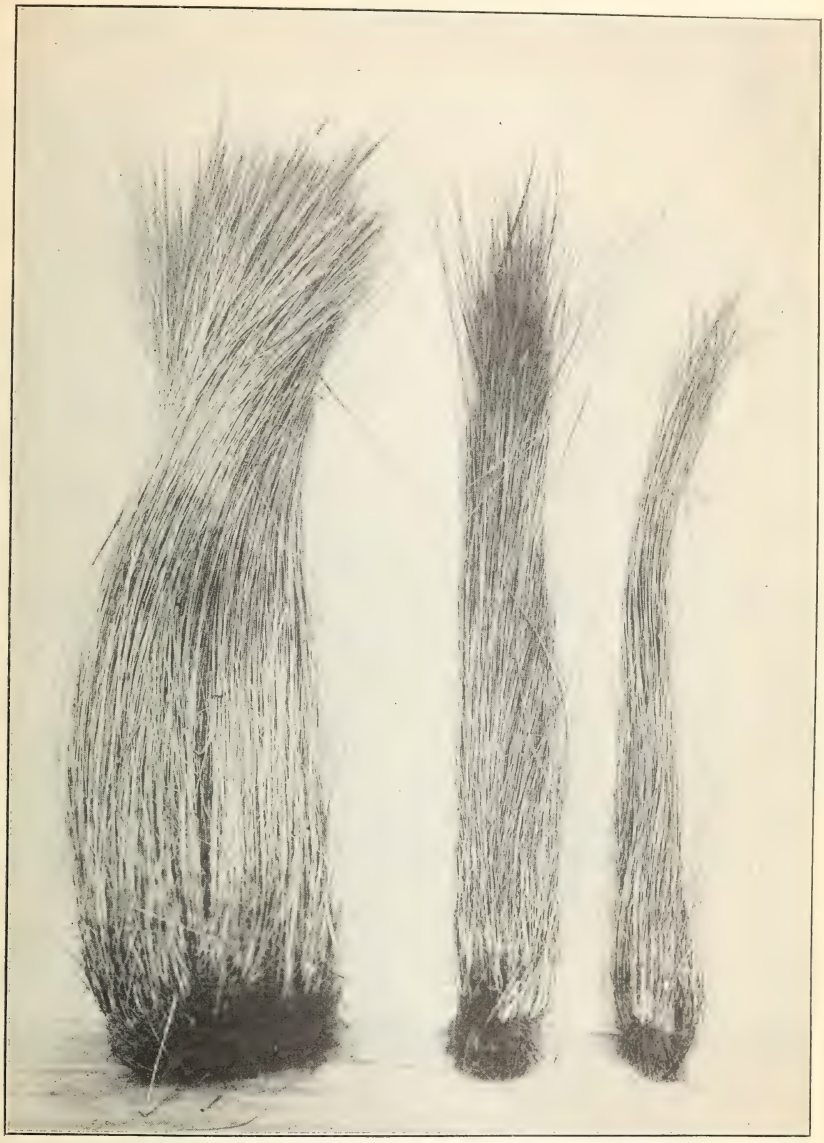

FIG. 7.—An average tuft and two small tufts of zacaton grass.

The writers have not as yet been able to obtain accurate figures as to the production of roots in pounds, but such figures as are available indicate a total production of from three to five million pounds per

annum. The cost of producing the roots per pound is, roughly, as follows:

Ground rent, or the harvesting privilege	$0.04
Harvesting	.01½
Cleaning and topping	.04
Hauling to primary shipping point	.00⅜
Freight to New York	.02

If the tops can be gotten out economically and reduced to pulp without an expensive freight haul, there would seem to be no question as to the promise of this material, which at present is purely a waste

FIG. 8.—A sparse stand of zacaton on the Vulcan de Agua, near Antigua, Guatemala.

product. It would be unwise to attempt to put a value on the tops delivered at a pulp mill, but it can be said that properly harvested esparto from Spain, Algeria, and Tripoli brings from $17 to $23 per ton in the English market. A good zacaton range can be profitably gone over for root brush material every third year.

LABORATORY TESTS OF PULP PRODUCTION.

An investigation of the paper value of any new fibrous plant can be conveniently separated into two distinct divisions, just as the manufacture of paper is commercially divided into two distinct branches. The first division embraces the separation and purifica-

tion of the plant-cellulose fibers, while the second division concerns the physical treatment and formation of these fibers into the finished sheet.

The separation and purification of the cellulose fibers is necessarily investigated first, and should be brought to as satisfactory a con-

FIG. 9.—Zacaton grass claiming a formerly cultivated field on a terraced hillside near Quezaltenango, Guatemala.

clusion as possible before actual paper making is considered. This investigation is most advantageously, almost necessarily, pursued in the laboratory, where conditions can be produced and accurately controlled. Fiber separation or production is effected commercially by one of the four following processes: The mechanical process, by

grinding; the sulphite process, by acid hydrolysis; the caustic-soda process, by hydrolysis with caustic soda; and the sulphate process, by hydrolysis with sodium hydrate and sulphid.

From previous general knowledge of the paper value of fibrous plants and from the similarity of zacaton to known species, it was decided to investigate this material by the soda process, which consists in subjecting the material to the action of a caustic-soda solution at moderately high temperature for a definite length of time, which operation is technically known as cooking. Since the soda solutions at the temperatures required exert a steam pressure of 50 to 100 pounds to the square inch, the cooking is effected in large, strong steel cylinders, known as digesters, which are of two general types, the upright stationary and the horizontal rotating. The preliminary cooking of zacaton was conducted in an autoclave, simulating the conditions of the upright stationary digester. The autoclave was of the regular laboratory type, of 7¼-liter capacity, composed of a rigid stand supporting the spun-copper autoclave shell, which could be securely closed by clamping on a bronze head or cover, the seal being secured by polished surfaces between the body and the head. The head was provided with a pressure gauge and thermometer well. A gas burner underneath the shell served to heat the charge to any desired temperature or pressure.

The method of operation is to place a certain weight of material in the body, to cover with a soda solution containing sufficient caustic soda to completely reduce the material, to securely close the autoclave, and, by means of the gas burner, to heat the entire charge to a definite temperature or pressure. This pressure is maintained the required number of hours, after which the charge is allowed to cool and the contents are removed and washed free from the dark-colored spent soda solution known as black liquor. Undercooked pieces of grass, which are invariably present, are separated from the pulp by screening through a No. 10 screen, in which the slots are 0.01 of an inch wide.

If the material under examination contains pith cells which by reason of their high percentage or quality tend to impart undesirable qualities to the finished sheet, it will be necessary to separate them from the true fiber at this point. Separation can readily be accomplished by manipulating the pulp on a 60 or 70 mesh wire cloth with a stream of water, whereby the small pith cells are washed away, leaving the long, true fibers on the wire. In the case of zacaton it does not appear necessary or advisable to separate the pith, and it was done only in cook No. 1.

In autoclave cook No. 1, 404 grams of grass, bone-dry basis, were treated with 24.4 per cent of caustic soda at a concentration of 19.7

grams of caustic soda per liter for 6 hours at a steam pressure of 90 pounds per square inch. It was very apparent that the long fiber, and even the screened fiber, was superior in quality to that of many other fibrous plants which had been tested, the soft feeling and bright luster being very noticeable.

In cook No. 2, 400 grams of grass were treated with 23 per cent of caustic soda at a concentration of 29 grams per liter for 7½ hours at a steam pressure of 90 pounds per square inch. The yield and general appearance of the fiber from this cook were very similar to those of the one preceding, and the conditions of treatment were possibly all that could be desired with this type of digester. Further economy of

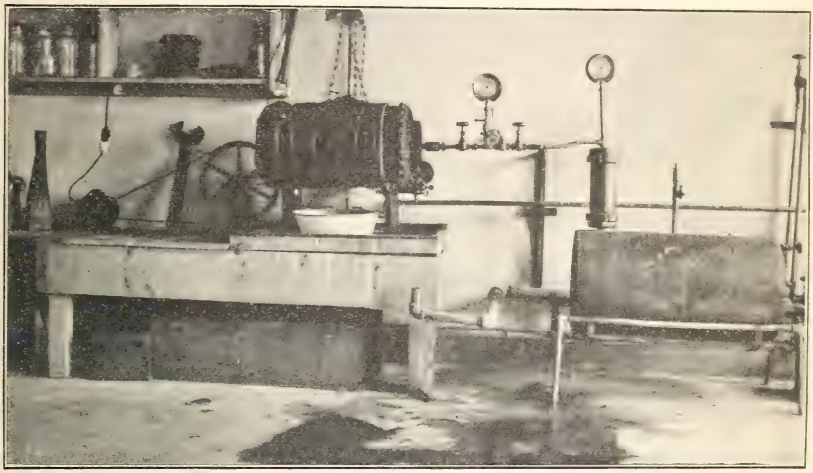

FIG. 10.—Experimental rotary pulp boiler.

treatment might be obtained with the rotary type of digester, where a lower percentage of caustic soda and a smaller volume of solution in proportion to the grass used would be possible. This method gives a more uniform pulp and affords a somewhat cheaper recovery of the spent soda, which is essential to the economic application of the soda process.

The rotary type of boiler employed was a ¼-inch steel shell, 12¼ inches in diameter by 29½ inches in length (38 liters in capacity), supplied with a hand hole and screw cap and mounted so as to rotate on its long horizontal axis one revolution per minute. (Fig. 10.) The charge was heated by gas burners underneath and controlled by a thermometer inserted in a well extending from the end of the shell into the center of the charge. Table I shows the yield of fiber obtained from these two cooks.

TABLE I.—*Fiber yields from two bone-dry cooks of zacaton grass, using different concentrations of caustic soda under a steam pressure of 90 pounds to the square inch.*

Cook.	Quantity.	Caustic soda.		Time boiled.	Pressure.	Yield.				
		Per liter.	Percentage.			Screenings.	Screened fiber.	Pith.	Long fiber.	Total fiber.
	Grams.	*Grams.*		*Hours.*	*Pounds.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
No. 1	404	19.7	24.4	6	90	4.7	35.4	7.5	27.9	40.1
No. 2	400	29.0	23.0	7½	90	4.6	37.1	.....	.....	41.7

Cook No. 3 was made in the rotary by treating 2,400 grams of grass, bone-dry weight, with 19 per cent of caustic soda at a concentration of 71 grams per liter for 6 hours at a steam pressure of 80 pounds per square inch, which gave a yield of 50.8 per cent of total fiber. The pulp was very uniform, soft, and of good appearance, and the cooking conditions were not as harsh or expensive as those for commercially treating poplar wood.

Cook No. 4 was made from a bale of zacaton which was received in a dry but very moldy condition, and the grass was quite brittle. A charge of 2,400 grams was treated with 19 per cent of caustic soda at a concentration of 70 grams per liter for 6 hours at a steam pressure of 80 pounds per square inch, giving a yield of 49 per cent of total fiber. It was impossible to distinguish any difference in quality between the pulp from the sound and that from the moldy grass, the fungi apparently attacking only the less resistant forms of hemicelluloses.

Cook No. 5 was made by treating 2,000 grams of zacaton with 18 per cent of caustic soda at a concentration of 70 grams per liter for 5 hours at a steam pressure of 90 pounds per square inch, giving a yield of 44.6 per cent of total fiber.

Cook No. 6 was made by treating 2,400 grams of grass with 16 per cent of caustic soda at a concentration of 70 grams per liter for 5½ hours at a steam pressure of 90 pounds per square inch, giving a fiber yield of 47.7 per cent of the total bone-dry grass. The expenditure of soda and time was very moderate, the yield was very fair, and the general appearance of the pulp was remarkably good.

MICROMEASUREMENTS OF FIBER AND OTHER CELLS.

Table II shows the comparative measurements of the cells of zacaton grass. The measurements were made on the screened stock from which paper No. 76 was made.

TABLE II.—*Comparison of the cell measurements of fibers and other cells of zacaton grass.*

Measurement.	Parenchyma of pith.		Long epidermal cells.		Parenchyma.		Bast.	
	Length.	Width.	Length.	Width.	Length.	Width.	Length.	Width.
	Mm.	*Mm.*	*Mm.*	*Mm.*	*Mm.*	*Mm.*	*Mm.*	*Mm.*
Maximum	0.112	0.072	0.079	0.011	0.223	0.058	3.0	0.013
Minimum	.072	.057	.072	.011	.162	.018	.5	.005
Average	.092	.061	.075	.011	.193	.038	1.7	.0085

The true fiber (fig. 11) has remarkably good felting qualities, but its length is less than that of esparto, which varies from 1.5 to 1.9 millimeters.

CHEMICAL INVESTIGATION OF THE GRASS AND PULP.

In cooperation with the Department of Commerce, an investigation of the chemical nature of zacaton grass and pulp in regard to such points as have a bearing on their paper value was conducted at the Bureau of Standards.[1]

The pulps examined were from cooks 7, 8, 9, and 10. The report of this chemical investigation, which needs no comment, is given in full, as follows:

1. *Original straw.*—(*a*) Ash; (*b*) moisture; (*c*) ether-alcohol extract; (*d*) water extract; (*e*) cellulose by (1) Cross and Bevan chlorin method, (2) Cross and Bevan dilute nitric method, (3) Renker's chlorin method; and (*f*) loss on boiling with dilute caustic soda.

It was found necessary to grind the straw very fine, on account of the lack of homogeneity of the samples. The largest samples practicable with the unground straw gave widely varying results, so recourse was had at once to grinding all the straw in a clean coffee mill before analysis.

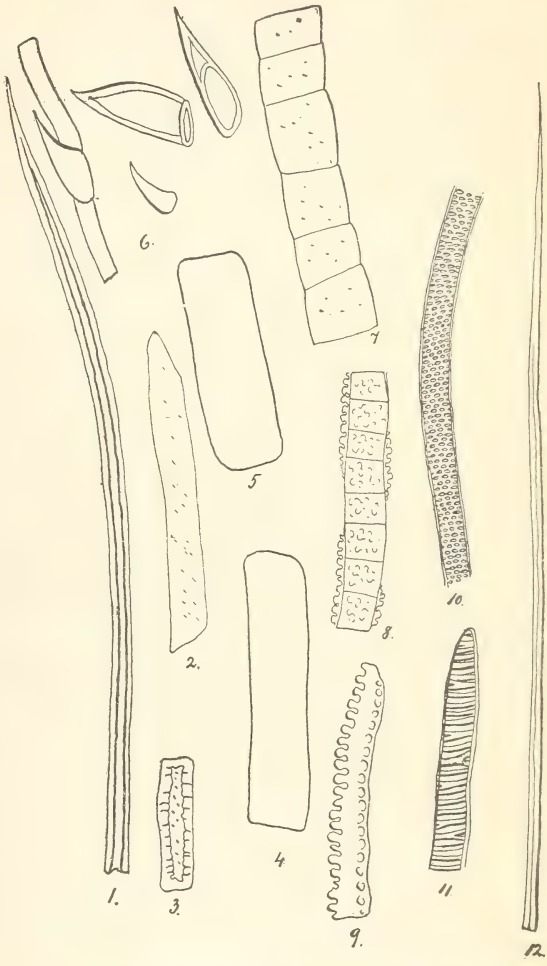

FIG. 11.—Pulp of *Epicampes macroura*, × 352: *1*, Bast; *2*, porous parenchyma; *3*, sclereid; *4* and *5*, parenchyma; *6*, modified epidermal cells; *7*, parenchyma of pith; *8*, short epidermis cells; *9*, long epidermis cells; *10*, pitted trachea; *11*, annular trachea; *12*, bast.

Analyses for comparative value should refer to dry material; hence the moisture was determined first to be 4.8 per cent. The ash gave 9.1 per cent in one determination and 8.8 per cent in another. In both cases a very large proportion of the ash was silica, as shown

[1] The chemical work on zacaton was done by Mr. George S. Tilley, at that time cellulose expert at the Bureau of Standards, United States Department of Commerce.

by volatilizing with hydrofluoric acid. In one case silica was 7.5 per cent of the original straw and in the second 6.5 per cent.

(*a*) This is a rather high ash content and a particularly large amount of silica, in comparison with the analyses of other straws published by Mayer, Müller, Hofmeister, and others. It fuses in a glass with the other constituents of the black liquor if sharply ignited, but on slower ignition gives a cake which easily falls to pieces on extraction with water. There will probably not be silica enough to interfere with the usual soda recovery on the large scale, especially if some of the English silica-removing processes are used. A trial of this point is, however, advisable where so much silica is found. This has, for example, nearly five times as much silica as esparto grass, as analyzed by Müller.

(*c*) Ether-alcohol extraction was carried out, not so much for the purpose of securing very valuable information, but in order to note abnormality, if any existed. One analysis gave 0.8 per cent loss, and a second 0.9 per cent, the nature of the extract not being further investigated. It is usually reported as fat, wax, and chlorophyll.

(*d*) The ether-extracted straw was further extracted for 14 hours with water in the modified Wiley extractor, which extracts at the temperature of the boiling solvent. The loss under this treatment was 5 per cent in one case, and 5.6 per cent in the second. As straws go, this shows a high degree of resistance, it being possible to extract as much as 70 per cent of some straws in this way. This value for Epicampes puts it between rye and wheat straw, roughly speaking, although of course there are wide variations in individual cases.

(*e*) "Cellulose" was determined by three methods; in each case the ash in the resulting product was determined and the ash-free white fiber resulting from the process followed was reported as "cellulose." No process now in use can claim to give a "normal cellulose" from straw or wood, and the significance of the per cent of "cellulose" reported is always relative to the analytical method followed.

(*f*) According to the original Cross and Bevan [1] method, as described in their book, the yield of "cellulose" averaged 41 per cent. Renker, in his recent book,[2] advises omitting the treatment with dilute alkali both before and after chlorination. The yield of fiber by this method was 51 per cent average. Cross and Bevan claim incomplete removal of lignin by this method, but Renker is certainly right in saying that the dilute alkali attacks the cellulose considerably. As a test of this, weighed samples of the straw were boiled 50 minutes with 1 per cent caustic soda, then filtered, dried, and weighed. The losses averaged 45.1 per cent on the bone-dry straw, with a residue of 54.9 per cent. This certainly leaves little for the chlorin to do in bringing the residue down to the 41 per cent found. It is probable that the true value lies between the two. Concordance in results may be obtained by either method, but of course adds nothing but confirmation of care in performance of the work.

The method of heating for 7 hours at 70° with 10 per cent nitric acid gave white residues (ash free) averaging 39.3 per cent. This is no doubt a minimum value and agrees fairly well with the results of the drastic original Cross and Bevan procedure.

2. *Blow-pit stock.*—Loss on bleaching was determined on the long-fiber unbleached stock. The determination is necessarily crude, because (1) if the stock be dried the chemical nature of the cellulose is changed and the bleaching may have a widely different effect, and (2) the water in the undried stock is about as serious a source of error. Determinations were made on both dried and undried stock. As nearly as possible, average samples of the undried stock were taken and moisture determined in some, while others were bleached.

Limit cases were taken with (*a*) strongly alkaline bleach, with much active chlorin; (*b*) slightly alkaline, with a small amount of free chlorin. With the stronger bleach

[1] Cross, C. F., and Bevan, E. J. A Text-Book of Paper-Making. 411 p., illus., 2 fold. pl. New York, 1907.

[2] Renker, Max. Ueber Bestimmungsmethoden der Zellulose. Aufl. 2, 107 p. Berlin, 1910.

the losses ran about 20 per cent, while with the more dilute they were from 8 to 10 per cent, with, as was noted, a large factor of possible error. The ordinary bleaching practice would give results nearer the lower limit, if anything.

The blow-pit stock from which the pith had not been separated did not bleach to a good color, even on long treatment. The loss in weight in one case which was weighed was 12.5 per cent, but the stock was not up to a pure white—rather a light cream color. This was due partly to the depth of coloring of the cooked pith and its resistant nature. The strongly resistant nature of the pith is seen by comparison of the results of dilute alkali and acid treatment with their effect on the original straw.

The loss on boiling the pith 1 hour with 1 per cent caustic soda was 18.5 per cent in one case and 17.8 per cent in another. The straw lost 45 per cent under similar treatment, and the pure bleached fiber 20 per cent.

The loss on heating the pith 7 hours at 70° with 10 per cent nitric acid was 18.3 per cent in one case and 17.9 per cent in another, while the straw lost 61 per cent and the pure bleached fiber lost 10 per cent under the same treatment. The necessity of removing the pith, even at the expense of some fiber, is apparent. There was quite a little fiber in the sample of pith, as shown by shaking up a sample with an excess of water, but no method of quantitatively separating the two has as yet suggested itself, unless fractional levigation or filtration may succeed in some form later. Chemically, there is not enough difference to effect even an approximate separation.

3. *Black liquor.*—The black liquor resulted from cooking Epicampes straw in an autoclave at 90 pounds for 7½ hours, using 23 per cent caustic on the straw weight, at concentration of 23.9 grams per liter. It was investigated for specific gravity, percentage of dissolved solids, saccharine matter, nitrogenous matter, ash, and total organic matter.

The specific gravity of the liquor was 1.046. It had 9.9 per cent total solids dissolved by one determination, and 10 per cent according to a second. Of this, 2.89 per cent should be the original caustic soda (of course, somewhat altered by combination with silica, etc.). A determination of the ash of the evaporated residue corroborated this; 6.2 per cent of the residue burned away in the blast, leaving 3.8 per cent ash, the 6.2 per cent being, then, total organic matter. The amount of ash does not by any means account for the silica of the straw, even assuming that half of it is left in the finished paper. It is probable that much of it is loosened by the cook and is washed away as a fine suspension.

A Kjeldahl analysis of an evaporated residue showed 2.1 per cent "proteid," using the usual factor in calculation from the ammonia found; that is, there was about 0.2 per cent proteid in the liquor before evaporation.

The addition of mineral acid to the black liquor gave a voluminous precipitate, consisting of acid cellulose, lignic acids, and some silicic acid. The amount of the precipitate was 4.8 per cent of the black liquor, leaving, then, 1.4 per cent organic matter in the filtrate.

The filtrate was examined for sugars. In 100 c. c. of the filtrate only 33 milligrams of sugar were present, calculated to dextrose. Probably the remainder in solution was levulinic acid, humic acids, acid-soluble modifications of cellulose, and similar products of the breaking down of the complex substances in the straw, but their small quantity and lack of value after identification argued against the necessarily long and laborious task of isolating them, even if they could be identified.

Conclusion.—The result of an investigation like the present one on substances of this nature gives necessarily only a general impression rather than a summary of definite and precisely measurable constants. In general, it may be said that *Epicampes macroura* gives a commercially practical yield of rather unusually high-grade paper fiber, with no particular trouble to be expected except from pith and silica in the process of manufacture.

CELLULOSE FROM ZACATON.

The sample sent for examination was bleached pulp of *Epicampes macroura* in the form of waterleaf sheets. The purpose of the work upon it was to determine the nature of its cellulose as completely as possible.

First, qualitative tests were tried. (*a*) Fuchsin-sulphurous acid (Schiff's reagent) turned a bit of the pulp pink on standing a few minutes. (*b*) Sachsse's solution (alkaline HgI_2,2KI) was reduced on boiling with the pulp. (*c*) Fehling's solution was reduced on boiling with the pulp. (*d*) Vitz's methylene-blue test (boiling a bit of the pulp for 15 minutes with 0.5 per cent aqueous solution of methylene blue) dyed the pulp deep blue, which was not removed by long washing. A bit of pure rag paper was put in the dye at the same time, as a check. It dyed light blue and washed nearly white under the same conditions of treatment. (*e*) Phenylhydrazin acetate boiled with the pulp gave a yellow hydrazone. (*f*) Ferric ferricyanid (Cross and Bevan) gave a blue precipitate with the pulp on standing some minutes. According to Cross and Bevan, this reaction is a quantitative measure of lignin. Later work has shown, however, that oxycellulose gives the same precipitate. (*g*) On distilling from a suspension of the pulp in hydrochloric acid of 1.06 specific gravity and adding a phloroglucin solution to the distillate, a large amount of furfural phloroglucid was precipitated. These reactions show conclusively that the sample contained oxycellulose in considerable quantity.

As oxycellulose is quite sensitive to attack by many destructive agencies, it was of importance to determine how much was in this sample from *Epicampes macroura* in relation to other celluloses. In the present state of knowledge (which, unfortunately, is rather a state of ignorance) of the constitution of cellulose, it is impossible to say how much of any given sample is oxycellulose. However, it is easy to compare samples and determine which has more or less of it.

Accordingly, comparative quantitative determinations were made of (1) copper number and (2) furfural yield on the cellulose from *Epicampes macroura*, a bleached soda poplar pulp, and on cotton "cellulose" simultaneously. The "copper number" of Schwalbe is the number of grams of copper precipitated by boiling 100 grams of the pulp in question for 15 minutes with Fehling's solution, under certain definite conditions of concentration, heating, and so on. In the absence of other reducing substances it is an admirable measure of oxycellulose; hence, it was used here with a modified Low volumetric method of determining the copper instead of Schwalbe's slower and no more accurate electrolytic method.

It is at once evident that the Epicampes cellulose contains slightly more oxycellulose than the poplar pulp and much more than the cotton. (Contrary to the commonly accepted belief, the writers have found most cotton cellulose to contain a little oxycellulose, as shown by many tests.)

For further confirmation and comparison, simultaneous quantitative estimations of the furfural yield from the same cotton, poplar pulp, and Epicampes pulp were made, following the method of Flint and Tollens. This, of course, would not be applicable in the presence of wood gums or various other pentosans or furfurosans, but was here applicable because the other furfural-yielding groups were absent.

The numerical results of these two analyses are shown in Table III.

TABLE III.—*Copper number and furfural yield of samples of cotton, poplar pulp, and Epicampes pulp in two determinations.*

Sample.	Determination No. 1.		Determination No. 2.	
	Copper No.	Furfural.	Copper No.	Furfural.
		Per cent.		*Per cent.*
Cotton	1.25	0.40	1.31	0.50
Poplar pulp	4.26	10.01	3.82	10.00
Epicampes pulp	4.58	10.92	4.39	10.62

It is here again evident that Epicampes cellulose is close to poplar pulp in oxycellulose content and contains slightly more of it.

This being so, the Epicampes cellulose should be more sensitive to attack by hydrolytic and oxidizing agents. In verification of this, determinations were made (*a*) of the action of alkaline solutions and (*b*) of nitric acid on all three celluloses, using the same three of the preceding work. First, the action of a 1 per cent aqueous NaOH solution on the three celluloses was tried. Weighed samples were boiled 60 minutes with a large excess of the solution, then filtered, washed, dried, and again weighed.

This verifies, in so far, the prediction from the relative amounts of oxycellulose. Next, the action of concentrated NaOH solution was tried, according to the usual procedure for determining "loss on mercerizing." The agreement here is as good as can be expected of this reaction, because of the slow penetration and uneven action of the cold concentrated solution. It shows the same relation between the celluloses, although more roughly than preceding determinations.

Next, the action of nitric acid was tried, (*a*) using "nitrating acid" and determining "gain on nitration" in the usual manner and (*b*) using 5 per cent HNO_3 at 70° C. for 7 hours. The resistance to combined oxidizing and solvent action of the nitrating acids (equal parts of concentrated sulphuric and fuming nitric acids) is very evident in the predicted order. Table IV shows the numerical results of these tests.

TABLE IV.—*Action on cotton, poplar pulp, and Epicampes pulp of a concentrated nitrating mixture and dilute and concentrated solutions of sodium hydroxid.*

Sample.	Determination No. 1.			Determination No. 2.		
	Loss in NaOH, 1 per cent solution.	Loss in NaOH, concentrated solution.	Total yield per 100 grams in nitrating mixture.	Loss in NaOH, 1 per cent solution.	Loss in NaOH, concentrated solution.	Total yield per 100 grams in nitrating mixture.
	Per cent.	*Per cent.*	*Grams.*	*Per cent.*	*Per cent.*	*Grams.*
Cotton	2.0	7.9	177.7	1.4	9.1	180.7
Poplar pulp	12.3	15.4	159.0	12.1	18.2	[1]
Epicampes pulp	19.3	17.3	149.2	19.4	18.4	140.8

[1] Sample exploded during the process of drying.

Next, the action of hot dilute (5 per cent) nitric acid was tested. Weighed samples of poplar pulp and Epicampes pulp were heated for 7 hours at 70° C. with 100 times their weight of 5 per cent HNO_3, then filtered, washed, dried, and weighed. Because of the small amount of Epicampes pulp remaining, no duplicate could be made. The soda poplar pulp lost 6.1 per cent and the Epicampes pulp 10.2 per cent in the process.

This evidence was considered sufficiently complete with regard to the oxycellulose question. The behavior of Epicampes pulp with the usual solvents of cellulose was tried, with results as follows:

(*a*) Schweitzer's reagent dissolved all but the merest faint trace of the fiber.

(*b*) Concentrated sulphuric acid dissolved the fiber quite rapidly and completely, with a very faint darkening in color—not nearly so dark as straw celluloses usually give with sulphuric acid.

(*c*) Zinc chlorid in concentrated hydrochloric acid dissolved the fiber more slowly than did sulphuric acid, but quite completely.

(*d*) Zinc chlorid solution swelled the fiber, but dissolved it only slowly.

The percentages of ash and moisture were also determined, ash being 2.2 per cent and moisture 4.8 per cent. Both determinations are of minor importance, the ash qualitatively and quantitatively being dependent on the previous treatment of the pulp and the moisture on the atmospheric conditions. The fact that they were not extraordinary had, however, to be determined. The moisture was within 0.2 per cent of that of poplar pulp under the same atmospheric conditions, indicating again the close chemical relationship that has been evident throughout in the comparison of poplar and Epicampes pulps, as it is well known that different forms of cellulose have widely differing hygroscopicity. It is, in fact, more closely related to poplar pulp than it is to straw celluloses, like those of wheat or rye. These latter give from 12 to 14 per cent of furfural, for example, while Epicampes pulp gave a 10.8 per cent average, poplar giving 10 per cent. The resistance of Epicampes cellulose to destructive agents in general is correspondnigly higher than that of the usual straw celluloses.

It was next intended to make a methoxyl determination on the Epicampes pulp, but microscopic examination showed (1) lignified cells still present and (2) cells in bundles that are usually completely separated by cooking, although only a very little of either. A methoxyl determination would be unfair to the sample when thus undercooked, and so was not carried out.

Direct determination of cellulose by any of the accepted methods is obviously useless here, (1) because the pulp has already been through processes for lignin removal and (2) because the presence of oxycellulose renders impossible a determination of cellulose accurate to 15 per cent, as it is always attacked much more than lignin by the usual reagents. There is no existing accurate method for these conditions.

Carbon and hydrogen determination by combustion would add, perhaps, a little to the already present wealth of evidence of oxycellulose presence, but the relation of furfural yield to carbon percentage is so well known that the carbon could be predicted to a fraction of a per cent. This determination, therefore, seemed needless for the complete characterization of the Epicampes fiber.

Hydralcellulose was tested for during the "copper number" determinations, by Schwalbe's method, and found absent.

To sum up the net result of these determinations: *Epicampes macroura* bleached pulp is a natural oxycellulose closely related to poplar pulp in chemical properties and considerably superior to the usual straw celluloses in power of resisting chemical attack by destructive agencies.

SEMICOMMERCIAL TESTS OF THE PULP.

Having experimentally determined the cooking conditions and found them to be reasonable and satisfactory and also having determined that the chemical nature of the pulp was satisfactory, the work was continued on a semicommercial scale and was planned to include the actual manufacture of paper. There is a tendency on the part of many to discount the practical value of results obtained

experimentally on a small scale, and doubtless in many cases this attitude is justified. Experimentation on a small scale has its own valuable sphere of usefulness, but great care should be exercised in its commercial interpretation. Therefore, with a view to giving this material a more reliable or commercial paper value the work was continued on a larger scale, more nearly under mill conditions and at a place where the services of actual mill employees could be secured for the work.

The digester employed for this work was of the upright stationary type, measuring about 2 feet in diameter by 10 feet high and heated by direct steam. The cooked charge was "blown" in the regular manner into a blow pit of ordinary construction, where it was drained and washed free from black liquor. After screening in the regular manner the stock was bleached with mill bleach liquor in a beating engine and bleach chest, washed free from bleach residues, made into the desired furnish, and suitably beaten, after which it was run through a Jordan refiner and then to a Fourdrinier paper machine. The greater part of this whole work was performed by the regular mill employees.[1]

The material used for the four cooks of this test was dry, but had previously molded to some extent, being the same as that used in cooks Nos. 4, 5, and 6. Table V shows the cooking conditions and the yields of the four cooks.

TABLE V.—*Conditions of cooking and total yields of fiber of four cooks of zacaton grass.*

Cook.	Charge, bone dry.	Caustic soda added.	Concentration of caustic soda.	Cooking pressure.	Hours under pressure.	Yield, bone dry, of total fiber.
	Pounds.	*Per cent.*		*Pounds.*		
No. 7	175	20	41	100	5	38.6
No. 8	182	19	90	90	6	33.4
No. 9[a]	192	18	.....	90	5	44.6
No. 10[a]	186	16	.....	90	6	45.2

[a] The charge in the digester was covered with water and heated to a steam pressure of 50 pounds per square inch for 1 hour, after which the water extract was all drained off and the residue cooked in the regular manner.

The higher yield of total fiber in cooks Nos. 9 and 10 is due to the lower percentage of soda added and the fact that its concentration was lowered by the water remaining in the grass after extraction. The general appearance of the pulp of these four cooks was very similar; they were soft feeling, bulky, and had a very silky luster. Screening was done on a No. 10 cut screen, and in place of a pulp thickener the screened stock was run over the wet end of a Fourdrinier paper machine and taken off at the first press. This procedure left the stock in fine condition to be transferred to the bleach beater.

[1] Much assistance and information was furnished by S. D. Warren & Co., Cumberland Mills, Me.

Screened pulp from the four cooks was combined into one beater charge, heated to about 40° C., and after being thoroughly mixed with bleach liquor to the equivalent of 21 per cent of its bone-dry weight of commercial bleaching powder, was pumped to a bleach chest to exhaust. When the bleach was practically all consumed and the

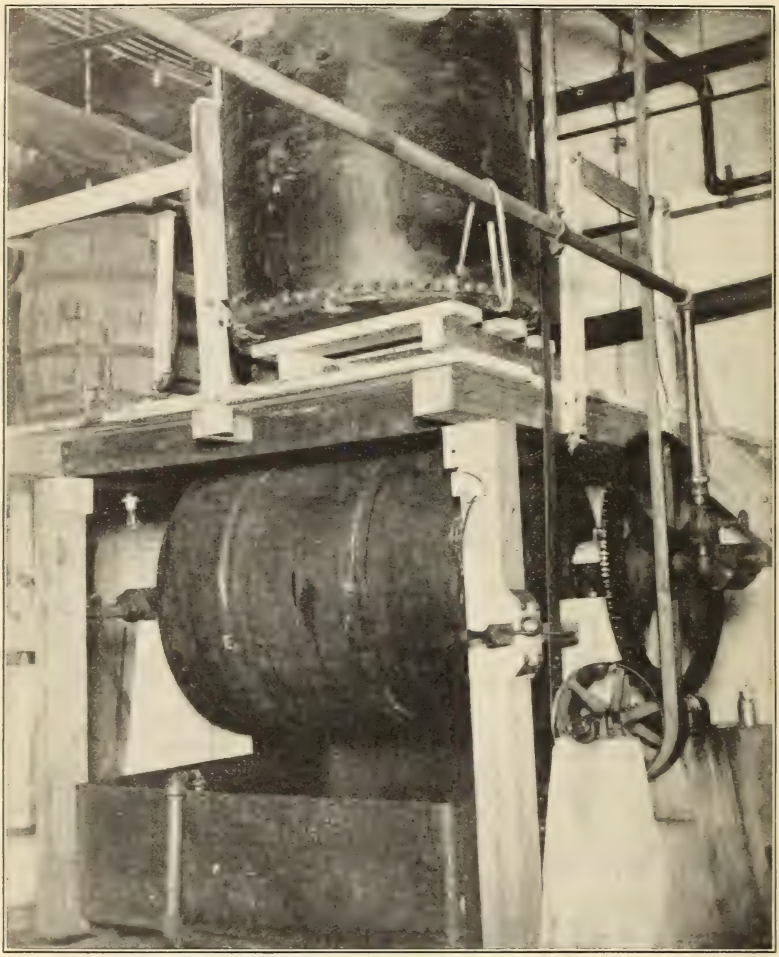

FIG. 12.—Semicommercial rotary pulp boiler.

color of the stock was found to be satisfactory, it was drained and washed free from bleach residues.

The bleached stock was then beaten with a medium brush for 2½ hours, loaded with 25 per cent of clay, sized with a 0.5 per cent resin size and 2 per cent of alum, and run through a Jordan refiner and then to a 30-inch Fourdrinier paper machine speeded to 90 feet per minute. The stock acted very well on the paper machine, giving

very little trouble and producing a machine-finished sheet of good appearance and quality. Physical tests on this sheet, designated as No. 41, are recorded in Table VIII.

Since the installation of a rotary type of digester by this bureau, further tests were made on Epicampes under the more favorable conditions afforded by this method of treatment. The rotary measured 6 feet in length by 4 feet in diameter, and was supplied with a large man head, thermometer well, pressure gauge, steam inlet, and steam relief through the hollow trunnions, and rotated at one-half a revolution per minute (fig. 12). A charge of 350 pounds could be handled conveniently, using a much stronger caustic solution and yielding a more uniform pulp.

The cooked charge was dumped into an iron drain tank underneath, where, by means of a false bottom, the fiber could be drained and washed with no loss. By means of a vacuum under the false bottom, the water could be drained from the fiber uniformly, leaving only 70 to 80 per cent of water in the fiber, in which condition it could be sampled and weighed with a good degree of accuracy. Uniformity of chemical action on the grass was very noticeable and assisted very much in the subsequent operations and quality of the pulp.

The grass used for these tests was of good, medium quality and in perfect condition. Four cooks were made, in order to secure sufficient pulp for a fair trial on the beating engine and paper machine, the cooking conditions of which are shown in Table VI.

TABLE VI.—*Conditions of trials of four cooks of zacaton pulp.*

Cook.	Grass.			Relation of caustic soda added.	Strength of caustic solution.	Caustic soda added.	Caustic solution added.	Maximum temperature, boiling.		
	Air dry.	Bone dry.						Time to reach.	Heat used.	Time held.
		Per cent.	Pounds.							
	Pounds.			*Per ct.*	*Per cent.*	*Pounds.*	*Gallons.*	*Hours.*	*° C.*	*Hours.*
No. 11	341	90.7	309	19.0	9.18	58.9	77	1¾	160 166	6
No. 12	361	91.0	328	20.1	9.18	66.0	86	2	160 166	4
No. 13	335	90.8	304	20.2	9.18	61.6	81	1¾	166	4
No. 14	342	90.8	311	20.3	9.18	63.2	82	1¾	166	3¾

As seen by the table, the cooking was controlled by temperature instead of by steam pressure. Since it is temperature and not pressure which induces chemical action, and since steam pressure is indirectly an expression of temperature, it is obviously correct to employ either for control. On account of the presence of more or less air in the steam supply, and on account of gases which are

liberated during the cooking, there is usually more pressure within the digester than the pressure corresponding to the temperature of the charge, and since this false pressure is usually unknown it seems preferable to employ temperature as the control.

The results of the tests are shown in Table VII.

TABLE VII.—*Results of trials of four cooks of zacaton pulp.*

Cook.	Fiber.			Relation of bone-dry fiber.	Causticity of waste solution.	Caustic.		
	Wet.	Bone dry.				Consumed by grass.	Relation of excess.	Relation of consumed to added.
		Per cent.	Pounds.					
	Pounds.			*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
No. 11	686	22.8	156	50.4				
No. 12	815	20.0	163	49.7	35.3	12.0	8.1	59
No. 13	715	20.1	143	47.0	38.2	11.5	8.7	56
No. 14	730	19.8	144	46.3	38.7	11.4	8.9	56

The average yield of fiber at 90 per cent bone dry is 43 per cent of the air-dry grass, which would average 80 per cent bone dry.

The excess caustic soda, 8.1 to 8.9 per cent of the bone-dry grass, is doubtless higher than is necessary, and it would indicate that less soda could have been employed in the cooking.

Pulp charges from the four cooks were remarkably similar in appearance and feeling; they drained and washed with great ease, a property not shared by the pulp of many plants and a fact of great importance in a commercial sense, because of the necessity of washing out and recovering the spent soda.

On screening the pulp charges through a No. 10 cut screen there remained undercooked screenings to the extent of 2.05 per cent of the bone-dry pulp, or 1 per cent of the bone-dry grass. The screened stock bleached easily and to a satisfactory color with the equivalent of 12.7 per cent of commercial bleaching powder.

After washing free from bleach residues, the stock was given four hours' medium brushing in a beating engine (fig. 13) and furnished with 24 per cent of clay, 1.8 per cent of resin size, and 2 per cent of alum.

At the close of the beating operation the charge was whitened by the addition of a small amount of blue or blue and red color, to offset the residual yellow tint invariably present in all bleached stocks.

The finished stock was run through a Jordan refiner and to a 30-inch Fourdrinier machine speeded to 85 feet per minute. Although the stock was a little too "free" to get the maximum felting of the fiber it acted very well on the machine and gave a machine-finished sheet of good appearance and quality. Physical tests of this sheet, designated as No. 76, in connection with those of sheet No. 41 are given in Table VIII.

PHYSICAL TESTS OF ZACATON PAPERS.

It is impossible to grade these papers according to any recognized classification, since the fiber from the zacaton plant is not recognized as the equivalent of any commercial paper stock. From the fact that the chemical examination placed the fiber very close to soda poplar fiber it might be regarded as the equivalent of soda poplar in grading the samples according to any scheme of classification. However, since the endurance of these papers, as well as their behavior under manufacturing conditions, yet remains to be proved, it would hardly be justifiable at present to regard zacaton fiber as the equiva-

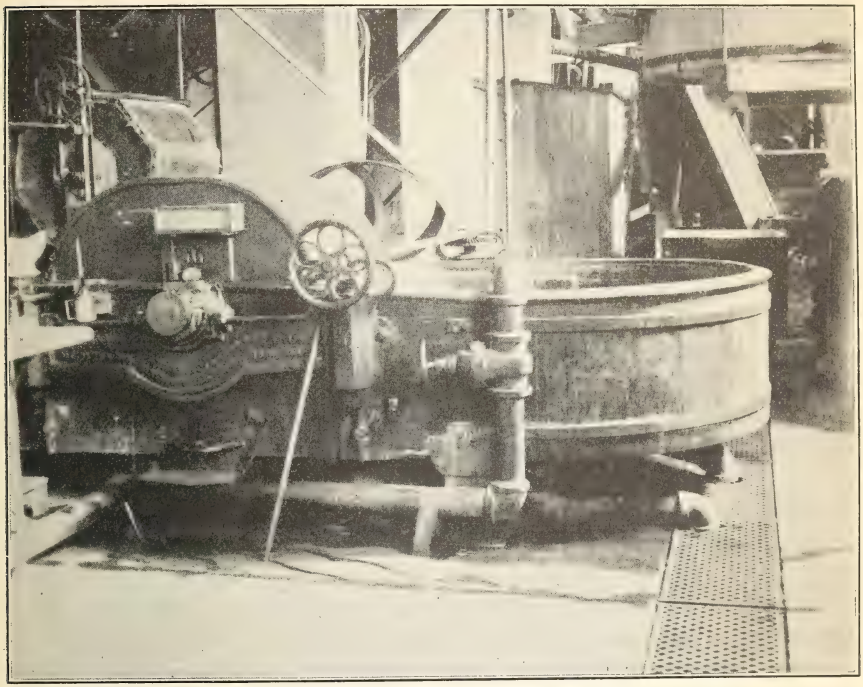

FIG. 13.—Beating machine supplied with washer.

lent of any commercial fiber. It would seem better, on the whole, to consider each physical test by itself in characterizing these special papers.

The system of classification and specifications followed in this work is that established by Veitch,[1] which in the main is used as the basis in the purchase of paper by the Government.

Sample sheets from the semicommercial tests were submitted to the Leather and Paper Laboratory of the Bureau of Chemistry for physical examination. This laboratory makes use of a testing room

[1] Veitch, F. P. Paper specifications. *In* U. S. Dept. Agr. Rpt. 89, p. 13–51, 4 fig. 1909.

which can be maintained accurately at a constant temperature and relative humidity, conditions which are absolutely essential to comparative and absolute paper testing.

The sample designated as No. 41 was manufactured from the combined pulps of cooks Nos. 7, 8, 9, and 10, and the sample designated as No. 76 was manufactured from the combined pulps of cooks Nos. 11, 12, 13, and 14. The report of the Leather and Paper Laboratory on these samples is shown in Table VIII.

TABLE VIII.—*Physical properties of six paper samples made from zacaton pulp, according to tests conducted by the Leather and Paper Laboratory of the Bureau of Chemistry.*

L. and P. No.	Mark No.	Ash.	Weight, 25x38, 500.	Bursting strength.			Strength factor, per wt., 25x38.	Thickness.	Folding endurance.		Folding factor.	
				Average.	Maximum.	Minimum.			Transverse.	Longitudinal.	Transverse.	Longitudinal.
		Per ct.	*Lbs.*	*Pts.*	*Pts.*	*Pts.*						
30612	41	19.5	51.0	28.0	29.0	27.0	0.55	36	27	42	0.53	0.82
30613	41	19.4	49.5	25.5	27.0	24.0	.52	34	25	39	.51	.79
30614	41	19.2	51.0	27.5	29.0	26.0	.54	37	38	35	.74	.69
30615	76	22.4	52.5	13.0	14.0	12.0	.25	40	4	3	.08	.06
30616	76	22.5	57.0	14.0	14.0	14.0	.25	38	4	3	.07	.05
30617	76	21.4	52.5	13.5	14.0	13.0	.26	39	3	3	.06	.06

Sample No. 41 contains the ash specified for a coated paper, namely, 20 per cent, although some uncoated book papers carry nearly this quantity. Practice varies in the different mills, some using more filler than others for the same general grade of paper. This sample shows a strength factor, which is the bursting strain divided by the ream weight, which is higher than that specified for a first-grade machine-finish printing paper, although the ash is four times that of this grade. The folding factor is likewise higher than is specified for a first-grade machine-finished printing paper.

Sample No. 76, according to the same classification, would fall below the fourth-grade machine-finish printing paper.

It should be noted here, however, that at the time the specifications of Report No. 89 were drawn up, the chief object was to secure better papers in general for the use of the United States Government. The specifications, therefore, are somewhat different and more rigorous than would have been the case had it been the intention to apply them to the general run of commercial papers.

According to present commercial usage, sample No. 41 would be regarded as much better than a first-grade machine-finish printing paper and No. 76 would be classed as a second-grade machine-finish printing paper

CONCLUSION.

Zacaton grass may prove to be a valuable paper stock, although at present it is a waste product and flourishes in a region remote from the paper-manufacturing sections.

The grass can be chemically reduced to paper stock by the soda process under less drastic and less expensive conditions than those employed for the reduction of poplar wood.

The well-known process, methods, and machinery employed for the manufacture of pulp from poplar wood are entirely suitable for the treatment of this material. In place of the wood-sawing, chipping, and screening machinery, a grass cutter, and possibly a duster, is required.

A production of 43 per cent of air-dry fiber from the air-dry grass is regarded as a very good yield, the fiber yield from poplar wood being from 46 to 48 per cent, and from esparto 43 per cent.

For bleaching the stock it has been found necessary to use more bleaching powder than in the case of poplar stock.

Paper manufactured from this stock has shown physical tests equal to those of a first-grade machine-finish printing paper.

The paper has a very satisfactory appearance and feeling. It is realized that in these two semicommercial tests the maximum possibilities of this material in all probability have not been attained, and better results may reasonably be expected. Moreover, an experienced mill organization after a few months of operation would learn the qualities of the stock and be in a position greatly to improve the product.

It would not be advisable, nor even possible, from the work here described, to make any estimate of the cost of manufacture or the value of the product. Such data can be secured only by extensive experimentation on a semicommercial scale or by actual mill operations.

PUBLICATIONS OF UNITED STATES DEPARTMENT OF AGRICULTURE RELATING TO MATERIALS FOR PAPER MAKING.

AVAILABLE FOR FREE DISTRIBUTION.

Suitability of Longleaf Pine for Paper Pulp. Department Bulletin 72.
Crop Plants for Paper Making. Bureau of Plant Industry Circular 82.
Pulp and Paper and other Products from Waste Resinous Woods. Bureau of Chemistry Bulletin 159.
Paper-making Materials and Their Conservation. Bureau of Chemistry Circular 41.
Paper Birch in the Northeast. Forest Service Circular 163.
Experiments with Jack Pine and Hemlock for Mechanical Pulp. Forest Service Miscellaneous.

FOR SALE BY THE SUPERINTENDENT OF DOCUMENTS.

The Grinding of Spruce for Mechanical Pulp. Forest Service Bulletin 127. Price, 15 cents.
Progress in Saving Forest Waste. Yearbook 1910. Price, $1.
The Utilization of Crop Plants in Paper Making. Yearbook 1910. Price, $1.

ADDITIONAL COPIES
OF THIS PUBLICATION MAY BE PROCURED FROM
THE SUPERINTENDENT OF DOCUMENTS
GOVERNMENT PRINTING OFFICE
WASHINGTON, D. C.
AT
5 CENTS PER COPY

▽

WASHINGTON : GOVERNMENT PRINTING OFFICE : 1915